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Fatigue life of layered metallic and ceramic plasma sprayed coatings

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Abstract

The application of thermally sprayed coatings can significantly enhance properties of coated parts such as thermal and wear resistance or biocompatibility. For example coatings prepared by HVOF are used for airplane landing gear parts and plasma sprayed coatings form heat shield on surface of turbine blades and vanes, several types of coatings are used in bone implants. In these application fields the fatigue behavior of coated components is of a paramount importance. The intrinsic properties of the deposited coating (modulus, microstructure, porosity etc.) play an important role. At the same time, the coating process can influence fatigue live through defects and residual stresses introduced to the substrate during spraying and associated preparation steps such as grit-blasting. The influence of substrate surface preconditioning and effect of individual layers of composite coatings on fatigue life were characterized. Plain substrates, grit-blasted substrates, and plasma sprayed specimens with one to three layers of coating were studies. The layered coatings were composed of alternating sequence of ceramic (Cr_2O_3) and metallic (Ni10wt%Al) layers. Deflection controlled resonance bending (R = -1) fatigue test of flat specimens was performed. The deformation amplitude was 1.3 milistrain in crack initiation site and loading frequency was around 80Hz. The significance of the effect of surface treatment on fatigue properties was examined statistically using Wilcoxon test. From the obtained data the effect of individual layers was deduced. In order to explain the observed fatigue behavior, the fractographic analysis and other means of coating and substrate characterization were performed. Strain hardening in substrate was characterized by micro/nanohardness measurement and EBSD analysis. Residual stress in substrate was measured using neutron diffraction.

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Keywords: functionally graded materials; fatigue life; neutron diffraction; grit blasting.

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1. Introduction

Thermally sprayed coatings can significantly enhance properties of coated parts - such as thermal and wear resistance or biocompatibility. Both ceramic and metallic coatings and their combinations are commonly used to achieve the desired surface properties. A classic example of ceramo-metallic composite is the thermal barrier coating (TBC) of yttria stabilized zirconia sprayed on top of NiCoCrAlY bondcoat, used at various parts of the aircraft engines. Another example is WC-Co wear resistant cermet coating used on airplane landing gear parts. Obviously, the fatigue behavior of both mentioned examples is of paramount importance. For better understanding of fatigue processes in these composite structures, the fatigue behavior of their ceramic and metallic components should be investigated. This can be accomplished by study the industrial composite coatings, or by testing simpler model composite coatings with different choice of ceramic and metallic components.

Many researchers investigated the fatigue properties of WC-Co and similar wear resistant coatings at room temperature, for an example see Ibrahim (2007). The mechanical fatigue properties of YSZ TBC's were studied by fewer authors, for example Rejda (1997) and Waki (2008). There are many other thermally sprayed materials that were fatigue tested, some of them in our laboratory. In the work of Musalek (2010) FeAl intermetallics tests are presented. Ti coatings for medical applications were studied by Cizek (2013), various metallic and ceramic deposits were tested by Kovarik (2008). In the papers on coating fatigue, the fatigue life of a coated part is attributed to the properties of the deposited coating such as its residual stress, elastic modulus, fracture toughness, hardness, adhesion, cohesion etc. At the same time, the coating process is expected to influence the fatigue life through defects and residual stresses introduced to the substrate during spraying, cooling and associated preparation steps.

The results obtained in our previous research on fatigue of metallic and ceramic deposits (see Kovarik (2004) and Kovarik (2008)) indicate that the effect of metallic and ceramic deposits on fatigue life differs significantly. The ceramic deposits lead to fatigue life increase in the majority of case, whereas the effect of metallic deposits strongly depended on the spray technology. Plasma spray and wire arc usually led to fatigue life decrease, whereas techniques with high peening stresses such as HVOF resulted in fatigue life increase. As an attempt to characterize the influence of ceramic and metallic layers in composite coatings on fatigue life, we performed a series of fatigue test of specimens at different phases of the coating process and characterized various properties of the tested deposits. The influence of grit-blasting, single material coatings and layered composites was tested. Two types of deposits were selected from Kovarik (2008), namely the fatigue life decreasing metallic deposit of Ni10wt%Al and fatigue life increasing ceramic deposit of Cr_2O_3.

The first step in coating application is grit blasting. It is used to clean and increase the roughness of the substrate. Abrasive particles are often implanted into the substrate during grit blasting. Together with the increased roughness, these particles act as stress concentrators. On the other hand, the impacting grit can induce compressive peening stress and deformation hardening below the substrate surface can improvement of the fatigue behavior.

During thermal spray deposition, significant heat is transferred to the substrate-coating composite and thermal gradients occur possibly leading to annealing of peening stress or to stress redistribution due to thermal mismatch stress and quenching stress. Both thermal mismatch and quenching stresses are superimposed on deformation induced stresses during fatigue test and may affect all stages of crack nucleation and propagation.

In this paper, we try to relate the fatigue life of ceramic and metallic coatings and their layered composites, to changes introduced during deposition as discussed above.

2. Experimental

The layered deposits were prepared at IPP, CAS, Prague, using a WSP PAL 160 torch. The substrate material was cold rolled sheet of mild steel (S235JRC, 4mm thickness), the as received specimens formed set P. The substrate was grit blasted by alumina particles prior to spraying, the grit blasted and not sprayed specimens formed set GB. Single layer coatings and layered deposits were prepared by alternating metal layers of Ni10wt%Al (denoted by M) and ceramic layers of Cr_2O_3 (denoted by C). Various combinations of sprayed M and C layers can be found in Table 2. The process conditions used for the deposition are summarized in Table 1.

The fatigue tests were carried out on an in-house developed resonance fatigue test device ”SF-Test”. The flat specimens with both sides coated were loaded by symmetrical ($R = -1$) cyclical bending (as cantilever beams) with a free end deflection amplitude fixed at u=4 mm at room temperature and with the loading frequency corresponding to the first natural frequency of the mounted specimens (70 - 100) Hz. The longitudinal strain at the crack initiation on the substrate surface was approximately 1.3×10^{-3} as indicated by strain gage measurement. The excitation frequency f_r was maintained using a phase locked loop technique. The crack growth in the specimens leads to decrease of resonance frequency. The frequency decrease is then used as a stopping condition of the experiment at approx. 30% cross-sectional damage. The corresponding number of fatigue cycles is denoted N.

Table 1. Deposition parameters for investigated coatings.

Parameter	Cr_2O_3 (C)	Ni10wt%Al (M)
Torch	PAL WSP® 160	PAL WSP® 160
Powder	Cr_2O_3, #2097	Ni10wt%Al
powder size	46-90 µm	100-140 µm
feed rate	300 g/min	417 g/min
spray distance	300 mm	350 mm
feed distance	32 mm	65 mm
Power	160 kW	160 kW

Table 2. Nomenclature and thicknesses of tested specimen sets, R_f denotes mean fatigue life relative to the as-received set.

Set	Description	coating thickness (µm)	N(cycles)	R_f
P	as-received	-	266487	1.00
GB	grit-blasted	-	229131	0.86
M	Ni10wt%Al-single	325	180756	0.68
MC	Cr_2O_3on Ni10wt%Al	98(M)+88(C)	293298	1.10
MCM	Ni10wt%Al on Cr_2O_3 on Ni10wt%Al	89(M)+140(C)+108(M)	254270	0.95
C	Cr_2O_3-single	598	383608	1.44
CM	Ni10wt%Al on Cr_2O_3	141(C)+113(M)	414308	1.55
CMC	Cr_2O_3 on Ni10wt%Al on Cr_2O_3	140(C)+199(M)+132(C)	417971	1.57

The coating microstructures were investigated on polished cross-sections. The fracture morphology was studied on fracture surfaces made available by fracturing the sample at cryogenic temperature. The JEOL JSM-840 (JEOL Ltd., Tokyo, Japan) scanning electron microscope (SEM) was used for the investigation of both fracture surfaces and microstructure. The energy dispersive X-ray microanalysis (EDX) was performed on SEM JEOL 5510LV equipped by IXRF 500 analyzer.

Phase composition of the deposits was investigated using X-ray diffraction (XRD). Vertical theta-theta diffractometer X'Pert PRO (PANalytical B.V., Almelo, The Netherlands) with Bragg-Brentano geometry and filtered CoKα radiation was used.

Strain hardening in substrate was characterized with help of Meyer hardness and EBSD. Meyer hardness was measured using NanoTest (Micro Materials Ltd., Wrecsham, U.K.) hardness tester with Berkovich indentor. Loading of 30 mN was applied. The indents were positioned on a line perpendicular to substrate-coating interface with 20 µm distance between the imprints. Electron back scatter diffraction (EBSD) study of substrate hardening was performed on a Jeol JSM-7600F SEM equipped with a HKL EBSD (Oxford Instruments PLC, Abingdon, UK) analyzer. The band slope (BS) signal was used. The BS signal represents electron backscattering pattern (EBSP) quality factor derived from the Hough transform that describes the maximum intensity gradient at the margins of the Kikuchi bands in an EBSP. High band slope signal means defect free homogeneous lattice with low number of defects. Strain hardening is a process connected with an increase of dislocation density and a decrease in the mobility of the dislocations in the adjacent material. It can thus be expected that strain hardened material will exhibit lower values of BS as compared to recovered material.

The residual stress depth profile in the substrate was measured using POLDI instrument at Swiss spallation neutron source (SINQ). The measurement of stress in the coating were also attempted, however the intensity of the diffracted signal was too low for residual stress determination. The Z-scan geometry as described by Ahmed (2008) was used with irradiated volume $19\times19\times d_{slit}$ mm^3. The height of the irradiated volume d_{slit} changed from 0.3 to 2 mm. The reflection on {110} planes was selected for strain estimation. Strain-free lattice spacing was determined from the measurement in the center of the specimen. The stress in longitudinal direction of the samples, which corresponded to the rolling direction, was computed by the relation $\sigma = \varepsilon(E_{110}/(1-\nu_{110}))$. Elastic modulus E_{110} and Poisson ratio ν_{110} were computed from elastic constants measured by Kim (2007).

3. Results

The microstructure of the investigated coatings is characteristic for thermally sprayed deposits and is illustrated in Fig. 1. The Cr_2O_3 layers contain splats of different flattening ratios and thermal history. Well flattened splats formed by impact of fully melted particles are observed as well as irregular equiaxial particles resulting from issufficiently melted or resolidified feedstock.

The Ni10wt%Al deposit is composed of Ni rich particles (brighter areas) and Al rich particles (darker areas). It is expected that significant amount of Ni and, especially, Al feedstock oxidized during the deposition. NiO was detected by XRD analysis. However, no reflections from phases containing Al were detected. According to the EDX analysis there is about 38 wt.% Al on the as-received surface and 23 wt.% Al on polished surface. The discrepancy between EDX and XRD supports the hypothesis that the Ni splats are partially covered by amorphous layer of alumina which is seen in XRD patterns only as amorphous halos.

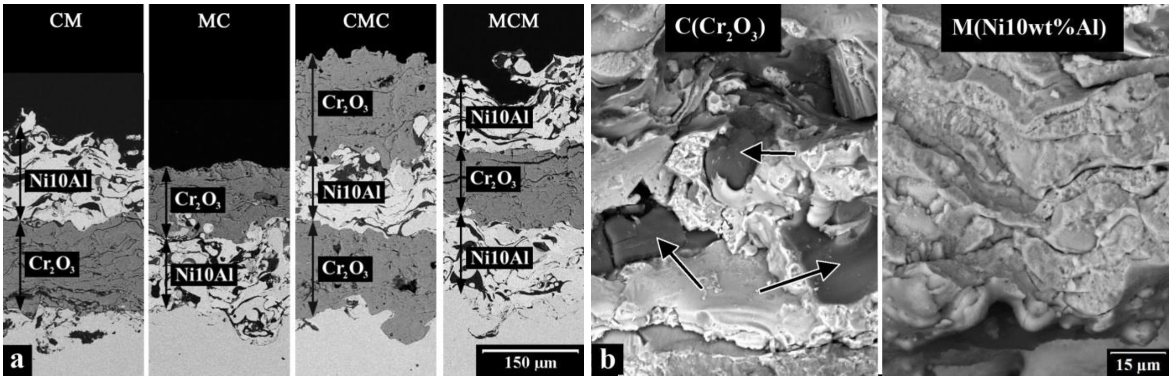

Fig. 1. (a) microstructures of investigated multilayer deposits observed using SEM in COMPO mode; (b) fracture surface of Cr_2O_3 (C) and Ni10wt%Al (M) layers; SEM in COMPO mode; dark areas marked by arrows exhibit increased concentration of Al and O

The results of fatigue tests are summarized in Table 2. The significance of surface treatment influence on fatigue life was tested by the Wilcoxon test. The results are summarized in Table 3. The numbers presented are probabilities (in %) of false rejection of “means are equal” hypotheses, i.e. low probability suggests significant influence of the surface treatment.

Table 3. Results of the Wilcoxon test; probabilities p (in %) of false rejection of "equal means" hypotheses; p > 5 % (i.e. possibly equal means) are highlighted.

	I.	II.				III.		
	M	**P**	**GB**	**MC**	**MCM**	**C**	**CM**	**CMC**
M	100.00	0.87	2.21	0.20	4.15	0.01	0.02	0.01
P	0.87	100.00	10.14	33.55	61.65	0.97	0.10	0.04
GB	2.21	10.14	100.00	5.20	43.20	0.02	0.01	0.00
MC	0.20	33.55	5.20	100.00	19.39	3.51	0.11	0.17
MCM	4.15	61.65	43.20	19.39	100.00	0.14	0.03	0.02
C	0.01	0.97	0.02	3.51	0.14	100.00	22.25	50.67
CM	0.02	0.10	0.01	0.11	0.03	22.25	100.00	97.37
CMC	0.01	0.04	0.00	0.17	0.02	50.67	97.37	100.00

The Wilcoxon test arranged specimen sets into three groups, with fatigue lives shorter (group I), similar (group II) and longer (group III) as compared to reference set P (see Table 3). The grouping of specimens in these three sets enables to draw a following conclusions from the fatigue test. Ceramic layer C increases fatigue life when applied on substrate and also on M layer. On the other hand, the M layer on substrate leads to fatigue life decrease, whereas it has no effect when applied on C layer. Therefore the C layers can protect the substrate from the detrimental influence of subsequent M layer. To suggest a mechanism which leads to the observed behavior of C and M layers, the results of the performed characterization have to be evaluated.

The fractographic analysis of specimens after final static cryogenic rupture was performed. The coating fracture morphology did not provide enough fractographical features for analysis (for illustration of coating fracture morphology see Fig1b). On the other hand, the crack initiation sites and crack propagation directions can be identified in the low carbon steel substrate despite the symmetrical nature of the fatigue loading. The imprints of grit blasted particles were often found to provide stress concentrators for crack initiation or re-initiation (in case the crack would start in coating). The cracks in the substrates grew by striation mechanism as 1/2 or 1/4 elliptical cracks. The cracks initiation sites location differs for the three observed specimen groups. For specimens in group I with fatigue lives decrease multiple crack initiations were located uniformly below the coated surface. The developed fatigue cracks were found in multiple parallel planes. This indicates significant number of stress concentration sites introduced by the coating process. For specimens in group II with neutral lives the cracks initiations were located close to specimen edges and the number of crack initiations was significantly lower. For specimens in group III with increased fatigue lives, there was only few initiations located mostly on uncoated surfaces. The coatings failed by mechanisms of intrasplat cracking and/or intersplat decohesion as described i.e. in Kovarik (2008).

The stress profile obtained by neutron diffraction is presented in Fig. 2. The compressive rolling stresses (see e,g, Dixit (1997)) were detected in as received specimens. The grit blasting process increased the magnitude of compressive stresses below the substrate surface. The deposition of the MC coating led to a decrease in compressive stress in the surface layer. The question is, however, whether deposition resulted in the peening stress annealing or whether it added additional tensile stress to the stress field. The stress redistribution in CM specimen as compared to GB specimens was detected, however the compressive stress with beneficial effect on the fatigue life was retained. The compressive stress below substrate surface appears as a necessary condition for fatigue life increase, however additional factors must be causing a difference between GB and CM fatigue lives.

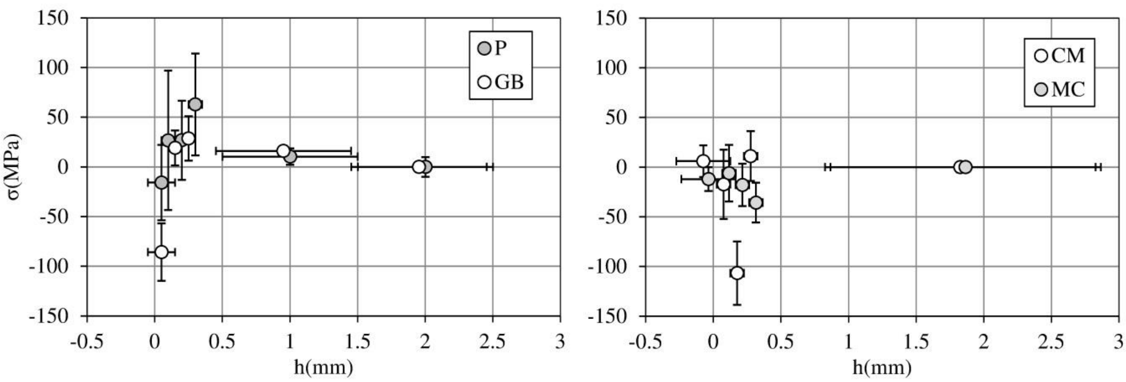

Fig. 2. Residual stress measured using neutron diffraction, vertical error bars mark the measurement error, horizontal error bats mark the measured area, i.e. the height of the neutron beam.

Fatigue resistance of the substrate depends also on work hardening introduced by the grit blasting. The obtained Meyer hardness profiles are summarized in Fig. 3 and clearly indicate work hardening up to a depth of approx. 70-100 μm. In contrast to residual stress, the hardened layer was preserved after the deposition for all coating types. In order to obtain higher depth resolution, an EBSD study was performed based on the band slope (BS) signal. The results of EBSD study are included in Fig. 3 and confirmed the profile obtained by microhardness measurement.

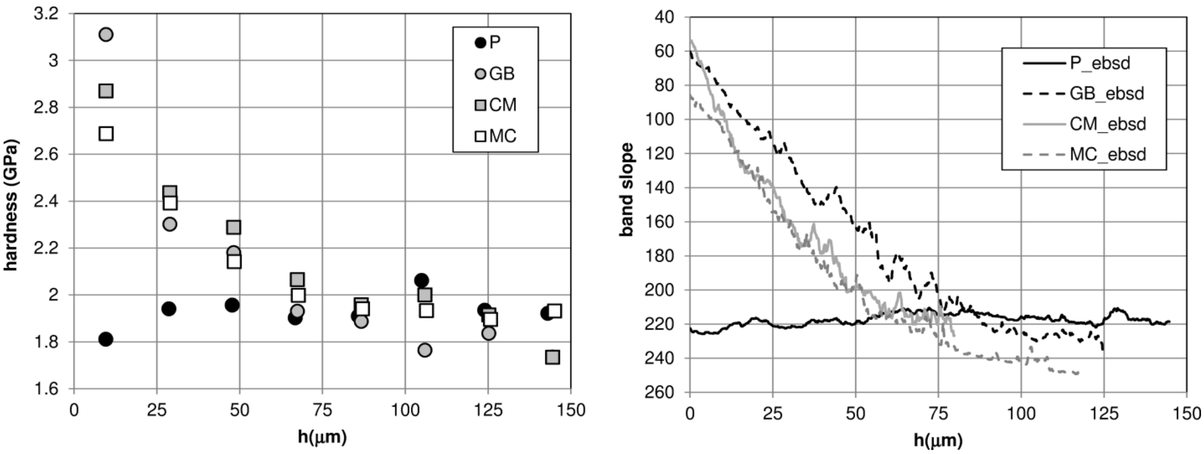

Fig. 3. Residual stress measured using neutron diffraction, vertical error bars mark the measurement error, horizontal error bats mark the measured area, i.e. the height of the neutron beam.

4. Conclusion

The influence of the investigated Cr_2O_3 and Ni10wt%Al coatings on fatigue life of coated bodies was analyzed. The fatigue test results can be related to other investigated properties as follows:

- Grit blasting did not influence fatigue life of substrate. The notch effect was compensated by compressive stress and strain hardening.
- The Cr_2O_3 deposit leads to a fatigue life increase. Strain hardened zone and compressive residual stress field are present in substrate below the Cr_2O_3 layer and can cause the observed fatigue life increase.
- The Ni10wt%Al coatings decreased fatigue life. The strain hardened zone was retained, but compressive stress was reduced in substrate. The increased number of fatigue cracks and their localization suggests notch effect of coating imperfections is additional factor reducing the fatigue life of Ni10wt%Al coated specimens.

5. Acknowldgement

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